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Title

# Method and Apparatus for Performing Chemical Reactions in a Plurality of Samples

This application claims the benefit of U.S. Provisional Application No. 60/407,899, which is incorporated in its entirety as a part hereof for all purposes.

## Field of the Invention

This invention relates to an apparatus for screening a plurality of sample materials for chemical activity, chemical equilibrium, and/or molecular transport.

## Background of the Invention

Screening candidate materials for chemical activity, for molecular transport, or for potentially catalytic properties is a time-consuming, labor-intensive process. Obtaining information concerning reaction rates at various compositions and process conditions, such as different temperatures and pressures, requires systematic investigation and the performance of many experiments.

An apparatus that could at least partially automate the process of simultaneously carrying out multiple reactions and simultaneously or sequentially making spectroscopic measurements to obtain information about reaction and molecular transport dynamics is considered to be advantageous. The present invention provides such an apparatus.

Summary of the Invention

This invention relates to a method and apparatus for simultaneously performing chemical reactions and simultaneously or sequentially making spectroscopic or other measurements on a plurality of samples, such as thin film samples. The apparatus of the present invention is capable of containing multiple samples in individual sample holding positions in a sample holder within a housing and maintaining those holding positions in chemical isolation from each other. Under control of a computerized controller, the apparatus positions the sample holder so that each sample holding position may be positioned adjacent to one or more ports connected to a distribution manifold. The apparatus exposes each sample to one or more fluids in liquid and/or gas phase, thereby carrying out a chemical reaction under controlled temperature, composition and pressure conditions. The sample holding positions may be positioned in a measurement station, such as an optical measurement station, within the housing so that the resulting chemical state may be characterized. Chemical reactions may be carried out within the measurement station and the chemical reaction and molecular transport dynamics may be monitored in real time.

Another embodiment of this invention is a method for testing a plurality of samples, by (a) simultaneously reacting all samples with a fluid, and (b) during the reaction of the samples with the fluid, subjecting each sample in sequence to analysis.

Yet another embodiment of this invention is a method for testing a plurality of samples, by (a) simultaneously reacting all samples with a fluid in a sealed vessel, and (b) after completion of the reaction of the samples with the fluid,

subjecting each sample in sequence to analysis in the sealed vessel.

A further embodiment of this invention is a method for testing a group of samples, by (a) simultaneously reacting all samples with a fluid in a sealed vessel, (b) before or after step (a), simultaneously reacting one or more members of a subgroup of the group of samples with a fluid in the sealed vessel, and (c) subjecting each sample to analysis.

A further embodiment of this invention is a method for testing a plurality of samples, by (a) bringing all samples to a predetermined temperature in a first chamber of a vessel, (b) simultaneously exposing each sample in a second chamber of the vessel, which is isolated from the first chamber, to a reactive fluid, and (c) subjecting each sample to analysis.

A further embodiment of this invention is a method for testing a plurality of samples, by (a) simultaneously exposing all samples to a non-reactive fluid in a first chamber of a vessel, (b) simultaneously exposing all samples in a second chamber of the vessel, which is isolated from the first chamber, to a reactive fluid, and (c) subjecting each sample to analysis.

A further embodiment of this invention is a method for testing a group of samples in a sealed vessel, by (a) placing one or more members of the group of samples in a position in the vessel to receive separate exposure to a reactive fluid, (b) simultaneously exposing those samples to the fluid, and (c) subjecting in the sealed vessel each member of the group of samples to analysis.

A further embodiment of this invention is an apparatus for testing a group of samples that includes (a) a fluid distribution system to simultaneously expose each sample to a reactive fluid, and (b) a holder for the group of samples slidable with respect to the fluid distribution system, and (c) an analyzer.

A further embodiment of this invention is an apparatus for testing a group of samples that includes (a) a fluid distribution system to simultaneously expose each sample to a reactive fluid, (b) an analyzer, and (c) a holder for the group of samples slidable with respect to the analyzer.

A further embodiment of this invention is an apparatus for testing a group of samples that includes (a) a fluid distribution system to simultaneously expose only the members of a subgroup of the group of samples to a reactive fluid, and (b) a holder for the group of samples slidable with respect to the fluid distribution system, and (c) an analyzer.

A further embodiment of this invention is an apparatus for testing a group of samples that includes (a) a fluid distribution system to simultaneously expose only the members of a subgroup of the group of samples to a reactive fluid, (b) an analyzer, and (c) a holder for the group of samples slidable with respect to the analyzer.

A further embodiment of this invention is a sealed vessel for testing a plurality of samples that includes (a) a fluid distribution system to simultaneously expose the samples to a reactive fluid, and (b) an analyzer in the sealed vessel that is isolated from the fluid distribution system.

A further embodiment of this invention is an apparatus for testing a plurality of samples that includes (a) a first chamber in which each samples is simultaneously exposed to a non-reactive fluid, (b) a second chamber, isolated from the first chamber, in which each samples is simultaneously exposed to a reactive fluid, and (c) an analyzer.

A further embodiment of this invention is an apparatus for testing a plurality of samples that includes (a) a first chamber in which each samples is simultaneously brought to a pre-determined temperature, (b) a second chamber, isolated from the first chamber, in which each samples is simultaneously exposed to a reactive fluid, and (c) an analyzer.

A further embodiment of this invention is an apparatus for testing a plurality of samples that includes (a) a holder for the samples, (b) a cover for the holder, and (c) an analyzer, wherein the cover is slidable with respect to the holder, and the holder is slidable with respect to the analyzer.

A further embodiment of this invention is an apparatus for testing a group of samples that includes (a) a fluid distribution system to simultaneously expose each sample to a reactive fluid; (b) a reaction chamber in which each sample is reacted with the fluid, the reaction chamber for each sample being separate and isolated from the reaction chamber for each other sample; and (c) an analyzer.

Brief Description of the Drawings

Figure 1 is a block diagram showing the elements of the apparatus of the present invention.

Figure 2 is a perspective view of the overall reaction apparatus of the present invention.

Figure 3 is an elevation view of the apparatus.

Figure 4 is a sectional elevation view of the apparatus, taken along section lines 4-4 of Figure 2.

Figure 5 is a sectional view taken along section lines C-C of Figure 3.

Figure 6 is a partial sectional view taken along section lines C-C of Figure 3.

Figure 7 is a first perspective view of the reaction assembly.

Figure 8 is a second perspective view, opposite the view of Figure 7, of the reaction assembly.

Figure 9 is a first sectional view of the reaction assembly.

Figure 10 is a second sectional view of the reaction assembly.

Figure 11 is a sectional partial view of the apparatus, taken along section lines 4-4 of Figure 2, showing the sample holder in the loading/unloading position.

Figure 12 is an enlarged sectional view of the apparatus, enlarging a portion of Figure 11.

Figure 13 is a sectional view of the apparatus, taken along section lines K-K of Figure 6.

Figure 14 is a view, partially in section, showing the sample holder in an optical measurement position.

Figure 15 is an enlarged view of a first embodiment of the sample holder.

Figure 16A is an enlarged view of a second sample holder having a sample hold-down clamp, the clamp being in a release position.

Figure 16B is a view of the second sample holder showing the sample hold-down clamp rotated to the holding position with the clamp in the up position.

Figure 16C is a view of the second sample holder showing the sample hold-down clamp rotated to the holding position with the clamp in the down position.

Figure 17 is a sectional view, taken along section lines 17-17 of Figure 16C.

Figure 18 is a sectional view, taken along section lines 18-18 of Figure 16C, showing an attenuated total internal reflection (ATR) measurement arrangement.

Figure 18A is an enlarged sectional view showing the interaction of light with the sample in the ATR measurement arrangement.

Figure 19 is a block diagram showing a main control routine for controlling the computer controller.

Figure 20 is a block diagram showing a control routine for controlling the spectrometer of an optical measurement system.

Figure 21 is a block diagram showing a routine for recording parameters and settings.

Figure 22 is a block diagram showing a routine for configuring elements of the system.

Figure 23 is a block diagram showing a routine for controlling valves and displaying set-points.

Figure 24 is a block diagram showing a routine for recording parameters and experimental data.

Figure 25 is a block diagram showing a routine for displaying spectral data.

Figure 26 is a block diagram showing a routine for controlling the positioning system.

Detailed Description of the Invention

In accordance with the present invention, a reaction apparatus containing a sample holder is arranged so that a plurality of samples to be reacted may be loaded into the sample holder, each sample being loaded respectively into a separate sample holding position in the sample holder. The sample holder is removable from the reaction apparatus to permit loading the samples in a controlled environment. When loaded, the sample holder may be inserted into an inner body of the reaction apparatus when the inner body is in a loading position. A mechanical detent assembly holds the sample holder in place in the inner body.

The sample holder, as carried within the inner body, may be loaded into the reaction apparatus through a loading/unloading section of the reactor apparatus. The loading/unloading section may be sealed with a manually installed cover. After the loading/unloading section is sealed with the cover, a gas control system is available to purge the loading/unloading section to eliminate any undesired gas within the reactor assembly.

Automated systems, as controlled by a computer, then set the parameters for a reaction, and cause the reaction to occur. A pressure control system may be commanded to bring the pressure and gas concentration in the reactor to a desired level. A temperature control system may be commanded to bring the temperature of the samples in the sample holder to a desired temperature, and a controller may command a fluid control system to introduce reaction fluid(s), which may be one or more gas(es) and/or liquid(s). A controller then commands a drive system to pull the inner body and the sample holder into the reactor housing into a fully inserted reaction position, and commands a positioning system to move the inner

body into a selected position within the reaction section of the housing.

A variety of sample holders may be employed. When the samples are analyzed by an optical method, an example of one type of suitable sample holder receives thin film samples mounted on either light absorbing, light transmitting or light reflecting substrates. The substrate may be planar or may contain a well to hold the sample. An example of a second type of optical sample holder receives samples mounted on a substrate, with an attenuated total internal reflection (ATR) crystal in contact with each sample, and has a clamping assembly that clamps the ATR crystal to the sample so that optical contact is maintained. Other kinds of sample holders may be used when other kinds of analytical measurements are made.

The protocol for the chemical reaction environment and the measurements are carried out under control of a control computer. Before the reaction begins, the sample positions may be flushed with an inert, non-reactive gas such as nitrogen. During the reaction phase, a positioning system moves the sample holder, held within the inner body, to a reaction position. The positioning system then moves the sample holder to an analytical monitoring section, and successively positions each sample at the correct position for analytical measurement during or, after completion of, the reaction. The arrangement for the desired type of analysis (i.e. the necessary equipment, commands and activating resources) is then engaged, and analytical measurement of each sample is performed to characterize the reacted sample. After measurement is completed, the sample holder is again brought to the loading/unloading section where, if necessary, the samples may be flushed with an inert gas, the temperature may

be raised or lowered to terminate the reaction, and the pressure returned to ambient pressure, such as to atmospheric pressure.

Figure 1 is a block diagram that illustrates the elements of the apparatus of the present invention. The system 10 contains a computer controller 20, such as an Optiplex GX1 from Dell Computer; an associated positioning system 30; a fluid distribution system 40; a temperature control system 60; a pressure control system 80; and a reaction apparatus 100. The fluid distribution system 40 may contain one or more electrically activated valves capable of controlling the passage of a fluid such as a gas or liquid, such as Swagelok model SS-4BG-3C gas valve, and associated tubing. The temperature control system may contain a commercially available temperature controller, such as a model CN3390 from Omega Corp. Stratford, CT , heating bands such as Type A heating bands manufactured by Watlow, Inc., and associated RTD temperature sensors, such as model DRW713237, and type J thermocouples, available from Technical Industrial Products. The pressure control system 80 may contain commercially available components such as a compressed gas supply, one or more electrically controlled pressure regulators, and electrically activated gas valves, such as Swagelok model SS-4BG-3C.

Figure 2 is a perspective view of the reaction apparatus 100 showing a generally cylindrical housing 120, an analytical monitoring section 160, and an attached a drive section 180. Figures 3 and 4 are side elevation views of the reaction apparatus 100 showing the cylindrical housing 120, which contains a loading/unloading section 130 having an airlock 132 and a cover 134; a reaction section 140; a distribution

manifold system 150; an analytical monitoring section 160; and an attached a drive section 180.

As seen in the perspective views of Figures 7 and 8 and sectional views 9 and 10, a reactor assembly 300 is shown, assembly 300 being contained within the housing 120, and being movable in a direction along the axis 120A of housing 120. The reactor assembly contains a cylindrical outer body 320 having a generally cylindrical bore 330 having an axis 330A and a plurality of ports 340. As seen in Figs. 3 and 4, the apparatus also contains heating elements 380, which may be one or more band heaters clamped around the reactor housing; and associated temperature sensing elements 390. As shown in Figs. 9 and 10, the outer body 320 contains a fluid distribution manifold 360. Bore 330 receives a slidable cylindrical inner body 400. A pair of constant tension springs 390, 392 bias the cylindrical outer body 320 and the cylindrical inner body 400 against the threaded drive screw 810. In an alternative embodiment, instead of using tension springs 390, 392, outer body 320, inner body 400 and sample holder 500 may all be made slidable in and out both ends of reactor assembly 300.

The inner body 400 has a generally cylindrical first bore 430 having an axis 430A, which is coincident with axis 330A, and a plurality of ports 440 (as shown in Figs. 11 and 12). First bore 430 receives slidable sample holder 500. The inner body 400 has a threaded second bore 450 that engages a threaded drive screw 810 (as shown in Figure 1) of the positioning system 30. As shown in Figs. 16A-16C, sample holder 500 has a plurality of reaction sample holding positions 504 for containing the samples to be reacted.

Referring again to Figures 7 and 8, the sample holder 500 is slidable along the axis 430A to a fully inserted position with the inner body 400. When the sample holder 500 is in the fully inserted position within the inner body 400, as seen in the sectional view of Figure 14, each of the plurality of sample holding positions 504 is aligned with each of the plurality of ports 340 of the outer body 320.

As shown in Fig. 1, the position control system 30 comprises the threaded screw 810, a drive motor 820 (such as a stepper motor) and associated reduction gears 830, a drive screw position encoder 840 and a drive controller 850 interfaced to the system controller 20.

When the ports 440 of the inner body 400 are aligned with the ports 340 of the outer body 320, a gas inlet passage 906 from the inlet distribution manifold to each sample holding position 504 is established; and a gas outlet passage 908 from each sample holding position 504 to the exhaust manifold 362 is established. This can be seen in sectional views Figures 9 and 10.

An example of one type of the analytical monitoring section 160 is the optical monitoring section seen in Figures 4, 5 and 6. It comprises a base assembly 600, at least one analytical ports (such as an optical port) 610, and at least one optical arrangement 640 (i.e. the necessary equipment, commands and activating resources for the particular type of optical analysis), such as a paired optical source 650 and detector 660 and an associated spectrometer 700 or 710. In the optical analysis, light may be passed from the optical source 650 to the optical detector 660 by reflection off of mirrors 662.

An optical arrangement 640 may, for example, be implemented using a spectrometer 700 (as shown in Figure 3) being capable of performing a measurement at ultraviolet or visible wavelengths of a sample contained on a sample holder positioned within a sample holding position 504 to characterize the sample. Alternatively a spectrometer 710 (also shown in Fig. 3) capable of performing a measurement at infrared wavelengths may be used to characterize the sample. The specific optical arrangement to be utilized is selected according to the characteristics of the sample. An optical transmission measurement 642, as shown in Figure 10, may be employed for samples that are at least partially transparent. An optical reflection arrangement 644, as shown in Figure 6, may be employed for samples that are opaque.

In one embodiment, an attenuated total internal reflection (ATR) arrangement 646, as shown in Figure 18A, may be employed for surface measurements of a sample S. The sample S is fixed either on the top or bottom by a rigid light conducting Attenuated, Total Reflection (ATR) transparent optical cover 530 such as a crystal. This assembly may be fixed by rigid supports 506, 508 on the top and bottom of the ATR crystal. The ATR crystal cross-section is preferred to be a trapezoid. Light L enters the ATR crystal normal to one of the end faces to make an angle of reflection with the faces F1, F2 that results in a total internal reflection condition. At each reflection there is emitted an evanescent standing wave, which decays exponentially with distance from the crystal interface into any material which is contacted with the ATR crystal surface. In Figure 18A, the top of the sample S is monitored within the evanescent waves at each reflection which transmit into the sample S. As the sample absorbs amounts of light within the evanescent waves, the absorption

can be detected from the light leaving the ATR crystal by a light detector.

Other types of analysis that may be used instead of, or in addition to, optical analysis include analysis selected from the group consisting of ultrasonic, electrostatic, magnetic, radio frequency or x-ray analysis.

In operation, the system 10 is capable of performing a plurality of chemical reactions. First, the sample holder 500 is loaded with samples to be reacted. When optical analysis, such as an ATR measurement, is to be made, a hold-down clamp 520, as shown in Figure 16A, is positioned in the release position so that a sample and the support 508 can be inserted into the sample holding position 504. The sample S, mounted on support 508, is inserted into the sample holding position 504, and a transparent optical cover 530 is placed over support 508, and top support 506 is placed over cover 530. The clamp 520 is rotated to the holding position with the clamp in the up position, as shown in Figure 16B. Then the clamp 520 is moved to the down position to hold the cover 530 tightly against top support 506, sealing the sample S in the sample holding position 504, as shown in Figures 16C and 18A. The sample holder 500 is inserted into the bore 430 of cylindrical inner body 400 of the reactor assembly 300 when the reactor assembly and the inner body are both positioned at an undocked position.

The cylindrical inner body 400 of the reactor assembly 300 is then moved to a docked position within the outer body 320 by the positioning control unit 30. At this time, the controller 20 may command the temperature control system 60 to bring the interior of outer body 320 to a predetermined temperature if necessary. The temperature control system 60

in such event energizes heating elements 380, and temperature-sensing elements 390 provide a feedback signal to the temperature control system 60. If pressure other than ambient is to be used, the control computer 20 commands the pressure control system 80 to either raise or lower the pressure within the apparatus to the desired pressure. Conventional pressure transducers (not shown) provide a pressure feedback signal to the pressure control system 80.

Next the controller 20 causes the fluid distribution system 40 to introduce one or more reactant fluid(s), such as gas(es) and/or liquid(s), to the samples within the sample holding positions 504, and the reactant fluid(s) react with the sample. When the reaction is complete, the positioning control unit 30 sequentially positions and re-positions the reactor assembly 300 so that each of the sample holding positions 504 is individually aligned with the analytical monitoring section 160. The sample holding positions can be positioned for individual alignment with the analytical monitoring section 160 in any order and more than once.

As each sample holding position 504 is brought slidably into its individual alignment with analytical port 610, at least one analytical measurement is made of that sample. Upon completion of the analytical measurements, the reactor assembly 300 is returned to the initial position adjacent the load/unload section 130. At this time the temperature and pressure within the apparatus is returned to ambient, if necessary. This may be facilitated by flushing the reaction assembly to quench the reaction, such as with an inert gas at ambient temperature and pressure. When the desired conditions have been reached, the inner body 400 of the reactor assembly 300 is moved to the undocked position, the cover 134 is

removed and the sample holder 500 is removed from the reactor assembly 300.

In various alternative embodiments, the invention provides a method for testing a plurality of samples, by (a) simultaneously reacting all samples with a fluid, and (b) during or after the reaction of the samples with the fluid, subjecting each sample in sequence to analysis. Once the airlock 132 is closed, the reaction of the samples with the fluid and the analysis are performed in a sealed vessel. While the samples remain in the sealed vessel, it is possible, if desired, to subjecting one or more of them to a second simultaneous reaction with a fluid, and a second analysis, and this sequence of steps may be repeated as many times as desired.

Each sample holding position 504 of the sample holder 500 provides a chamber in which the temperature or the pressure is controlled when the sample in that position is reacted. Each such reaction chamber is isolated from the reaction chamber provided by each other sample holding position. The isolation is provided by the fact that the sample holder 500 is slidable within the inner body 400, and the inner body is slidable with in the outer body 320. At any sample holding position at which there is a corresponding port in the inner body, when the inner body is moved such that the port in the inner body is aligned with the port in the outer body, the sample is exposed to the fluid in the manifold of the outer body. A reaction chamber exists, for example, when a port in both the outer and inner bodies are lined up with a sample holding position, and the ports have access to a fluid distribution manifold. That sample holding position is, however, isolated from all other sample holding positions and from the analytical port by the annulus of the outer body and

the annulus of the inner body. The invention thus provides a method in which the chamber in which each samples is exposed to or reacted with the fluid is isolated from the chamber in which each samples is subjected to analysis.

The analysis may be performed during, or after completion of, the reaction of the samples with the fluid.

In one segment of the reaction apparatus, when the ports in the inner body are aligned with the ports of the outer body, all sample holding positions are exposed to the fluid in the manifold, which may be a reactive or non-reactive fluid. In this segment, it is thus possible to simultaneously expose all samples to or react all samples with, the fluid. In another optional segment of the apparatus, however, a port in the inner body is not available for alignment with each port in the outer body. In this segment, it is thus possible to simultaneously expose one or more members of a subgroup of the samples to, or react one or more member of the subgroup with, the fluid. A subgroup of the group of samples in the sample holder is a number of samples that is less than the number in the whole group. The number in the subgroup may be one, or any other number that is less than the number in the whole group. The step of exposing or reacting the subgroup may be performed before or after the step of exposing or reacting the whole group.

The samples may be brought to a predetermined temperature in a segment or chamber of the reaction vessel before the sample holding positions in the sample holder have been placed in alignment with the ports in the outer body. The exposure or reaction of the samples may thus be conducted in a chamber of the apparatus that is isolated from a temperature-adjustment chamber by the sliding motion of the sample holder

moving into alignment with the ports in the outer body. When the sample holder is positioned in that alignment, moving the inner body such that its ports are also in the same alignment exposes the samples to the fluid in the manifold. After completion of reaction and analysis, the sample holder can be returned to the former position at which time the temperature of all samples can be further adjusted to a temperature above or below the predetermined temperature. In similar fashion, the samples may be exposed to a non-reactive fluid in a different segment of the apparatus from that in which they are exposed to a reactive fluid.

As mentioned above, the samples are placed in position to receive exposure to a fluid when the sample holding positions are placed in alignment with the ports in the outer body. Then by sliding the inner body component of the apparatus relative to the outer body component, an inlet passage is created for the fluid to flow from the manifold into the area of the sample holding position. In this sense, the inner body forms a cover for the sample holder with the result that the cover can be open when the ports of the inner body are in alignment with the ports of the outer body, and can be closed when the ports are not in alignment. When the sample holder is later moved into alignment with the analytical port, the sample holding position remains isolated by the annulus of the inner body from the reaction chamber previously formed when the respective ports of the inner and outer bodies were in alignment directly over the sample holding position.

After removal of the sample holder 500 from the reaction vessel, the sample hold-down clamp 520, if used, is released from down holding position to the up position (Figure 16C), and then the clamp may be rotated to the sample release position (Figure 16B) and the in the up position (Figure 16A).

Figures 19 through 26 depict, in block diagram form, software for controlling the system 10. Figure 19 is a block diagram showing a main control routine for controlling the computer controller. Figure 20 is a block diagram showing a routine for controlling a spectrometer when the analytical method employed is an optical measurement system. Figure 21 shows a routine for recording parameters and settings. Figure 22 shows a routine for configuring elements of the system. Figure 23 depicts a routine for controlling valves and displaying set-points. Figure 24 shows a routine for recording parameters and experimental data. Figure 25 depicts a routine for displaying spectral data when the analytical method employed is an optical measurement system. Figure 26 shows a routine for controlling the positioning system.

In operation, the system 10 is controlled by software that utilizes a graphical user interface to enable the user to operate the reaction apparatus 100 in an automated manner. The user is enabled to program all process, measurement and analysis parameters before the experiment is initiated. This programming is divided into three main stages: Set-Up, Experiment and Analysis.

In the Set-Up Stage, the user selects all process and measurement parameters. Process parameters include all temperature set-points for the temperature control system 60 for the loading, reactor and unloading sections; vacuum or pressure level for the pressure control system 80; motor drive controller parameters such as movement velocity; hold times for loading, preheat and unloading quench gas flows; as well as activation schedule to the fluid distribution system 40 for the solenoid-actuated valves which handle the loading-preheat fluid and unloading-quench fluid. When the

analytical method employed is an optical measurement system, the measurement parameters may include, for example, spectroscopy specifications for a UV/Visible spectrometer 700 and FTIR 710; identification of which sample positions 504 to measure; any desired delay time between sampling cycles; the total number of sampling cycles; and data storage path. All of these parameters completely define the experiment, and are recorded in a separate method file. The method file allows the user to document the experiment in a laboratory record, and may also be used as a template for future experiments.

The Set-Up Stage parameters are selected by the user by clicking on a "Set-Up" control button. This action makes available several additional control buttons that access different classes of experimental parameters. For example, a "Set Points" control button displays a window in which the user enters all temperature set points. A "Data Path" control button displays a window that allows the user to either define or specify an existing file system directory or create a new file system directory in which to store the experimental data files. A "Motor Sampling" button displays a window that permits the user to calibrate the motor 820, specify active sampling positions during the experiment, as well as report motion data from the drive controller 850. When the analytical method employed is an optical measurement system, a button such as an "Ocean Optics" button displays a window that permits the user to specify UV/Vis spectroscopy parameters for a spectrometer, such as an Ocean Optics spectrometer 700. A button such as a "Nicolet" button displays a window that permits the user to specify FTIR spectroscopy parameters for a spectrometer such as a Nicolet spectrometer 710.

A "Parameters" button displays a window that permits the user to program the experimental method and sequence. The

experimental method comprises sections entitled "Start", "Sampling" and "End". Each of these sections is optional and may be selected as either active or bypassed during the experiment. If the user activates the Start section, then the user may specify loading zone temperatures, loading fluid treatment flows and exposure time. If the user activates the Sampling section, the user may specify the number of sampling cycles, sampling kinetics as well as any delay time between sampling cycles. Furthermore, the user may specify the unloading temperature in advance of the End section so that the temperature may be adjusted by the temperature controllers during the experiment.

There are two types of sampling kinetics. In linear sampling kinetics, the user specifies a constant delay time between sampling cycles, which is maintained over all sampling cycles. In logarithmic sampling kinetics, the user specifies an initial delay time between sampling cycles. Here the delay time is kept constant for ten sampling cycles, and then doubled for the next ten sampling cycles. This process repeats until all specified sampling cycles have been followed. The logarithmic kinetics specification is ideal for reactions that are fast in the beginning, become progressively slower but ultimately last for long periods of time. Thus an optimal amount of data are collected and stored for the user to analyze. If the user activates the End section, the user may specify the unloading zone temperatures, unloading-quench gas treatment flows and exposure times.

In the Experiment Stage the user initiates the programmed instructions set in the Set-Up stage. Here the computer autonomously operates the reactor, and controls the process environment and data collection without further presence required of the user. The software does provide the user the

capability to pause and restart as well as to abort the experiment should such actions be required. The Experiment Stage is accessed by the user in the software by clicking on an "Experiment" control button in the graphical user interface.

In the Analysis Stage, when the analytical method employed is an optical measurement system, the user may employ utility subroutines that analyze the spectra series collected during the experiment. Individual IR, UV/Visible or other spectra may be accessed and analyzed independently. Alternatively, the user may select an entire series or a subset of a series to analyze in the identical manner. Such analyses typically involve selecting a baseline over a range of wavelengths, and then integrating the area within a spectral absorbance within another range of wavelengths. The spectral absorbances are normalized and recorded as a function of experiment time in a text data summary file. The text data file can be imported to suitable kinetics analysis software to derive rate expressions from the measured data. The Analysis Stage utility subroutines are accessed by the user in the software by clicking on a "Data Analysis" control button.

Examples of various other embodiments of this invention are described below. One embodiment of this invention is a method for testing a plurality of samples by (a) simultaneously reacting all samples with a fluid, and (b) during the simultaneous reaction of all samples, subjecting each sample in sequence to analysis. A further embodiment of this invention is a method for testing a plurality of samples by (a) simultaneously reacting all samples with a fluid, and (b) optically analyzing each sample using two or more optical methods, each method using light having a different wavelength in the range from about 190 nanometers to about 900 nanometers

or in the range from about 2,500 nanometers to about 25,000 nanometers.

A further embodiment of this invention is a method for testing a plurality of samples by (a) changing the temperature of all samples in a first chamber, (b) simultaneously exposing all samples in a second chamber, which is isolated from the first chamber, to a reactive fluid, (c) analyzing each sample, and (d) after completion of analysis, changing the temperature of all samples in the first chamber. The temperature of the samples may be changed by simultaneously exposing the samples to a non-reactive fluid, and the temperature of the samples may in any step be increased or decreased, such as by at least about 100°C. An exemplary non-reactive fluid is nitrogen.

A further embodiment of this invention is an apparatus for testing a plurality of samples that contains (a) a reaction chamber in which all samples are reacted with a fluid, and (b) an analyzer that performs two or more optical methods, each method using light having a different wavelength in the range from about 190 nanometers to about 900 nanometers or in the range from about 2,500 nanometers to about 25,000 nanometers.

In the above embodiments, during the testing procedure, the samples may be reacted with a fluid in a chamber in which the temperature or the pressure is controlled. The fluid may be one or more gases and/or one or more liquids. Before reacting the samples with the fluid in a second chamber, the temperature of all samples may be changed in a first chamber, the first chamber being isolated from the second chamber. The temperature of all samples in the first chamber may also be changed after reacting the samples with the fluid. The temperature of the samples may, for example, be increased

before the reaction, and decreased after the reaction, or vice versa. The first chamber may be isolated from the second chamber by sliding the sample carrier.

Another embodiment of this invention is an apparatus for testing a plurality of samples that contains (a) a fluid distribution system to simultaneously expose each sample to a reactive fluid, and (b) a transparent holder for one or more samples, and (c) an optical analyzer. Another embodiment of this invention is an apparatus for testing a plurality of samples that contains (a) a fluid distribution system to simultaneously expose each sample to a reactive fluid, and (b) a holder for one or more samples that comprises an attenuated total reflection crystal, and (c) an analyzer.

A further embodiment of this invention is an apparatus for testing a plurality of samples that contains (a) a first chamber in which all samples are simultaneously exposed to a non-reactive fluid, (b) a second chamber, isolated from the first chamber, in which all samples are simultaneously exposed to a reactive fluid, and (c) an analyzer. The non-reactive fluid or the reactive fluid may be a gas, and the non-reactive fluid may be nitrogen. A further embodiment of this invention is an apparatus for testing a plurality of samples that contains (a) a first chamber in which the temperature of all samples is changed by simultaneous exposure to fluid, (b) a second chamber, isolated from the first chamber, in which all samples are reacted by simultaneous exposure to a fluid, and (c) an analyzer.

A further embodiment of this invention is an apparatus for testing a plurality of samples, comprising (a) a first fluid distribution system to simultaneously expose all samples to a reactive fluid in a reaction chamber, (b) a second fluid

distribution system to individually expose each sample in sequence to a reactive fluid in a reaction chamber, and (c) an analyzer. A reactive fluid may be a gas, and the reactive fluids may be different. The different fluid distribution systems are accessed by placing the sample holding positions under different ports in the outer body that are served by different fluid distribution manifolds.

In all of the embodiments described above, the analysis may be optical analysis, such as passing light waves through a sample, or reflecting light waves from a surface of a sample. Two or more optical methods may be used if desired, each method using light having a different wavelength in the range, for example, of from about 190 nanometers to about 900 nanometers or in the range from about 2,500 nanometers to about 25,000 nanometers. All optical methods may be performed simultaneously, and the analysis may be conducted during a simultaneous reaction of all samples. Other useful methods of analysis include sonic, ultrasonic, electrostatic, magnetic, radio frequency or x-ray analysis.

Those skilled in the art, having the benefit of the teachings of the present invention as set forth herein, may effect numerous modifications thereto.